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INTERNATIONAL APPLICATION NO.
PCT/FR99/01652INTERNATIONAL FILING DATE
JULY 8, 1999PRIORITY DATE CLAIMED
JULY 9, 1998

TITLE OF INVENTION

GLAZING HAVING ELECTRICALLY CONTROLLABLE OPTICAL AND/OR ENERGY PROPERTIES

APPLICANT(S) FOR DO/EO/US

Philippe BOIRE, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☐ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 18 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
A **SECOND** or **SUBSEQUENT** preliminary amendment.
16. ☐ A substitute specification.
17. ☐ A change of power of attorney and/or address letter.
18. ☐ Certificate of Mailing by Express Mail
19. ☒ Other items or information:

Request for Consideration of Documents Cited in International Search Report

Notice of Priority

PCT/IB/304

PCT/IB/308

Drawings (1 Sheet)

Docket No. 1247-0855-0V PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

:

PHILIPPE BOIRE, ET AL.

: ATTN: APPLICATION DIVISION

SERIAL NO: NEW U.S. PCT APPLICATION
(Based on PCT/FR99/01652)

FILED: HEREWITH

:

FOR: GLAZING HAVING
ELECTRICALLY CONTROLLABLE
OPTICAL AND/OR ENERGY
PROPERTIES

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows.

IN THE SPECIFICATION

Please amend the specification as follows:

Page 1, before line 1, delete the title of the invention in its entirety, and insert therefor:

GLAZING WITH OPTICAL AND/OR ENERGETIC PROPERTIES CAPABLE OF
BEING ELECTRICALLY CONTROLLED

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IN THE CLAIMS

Please amend the claims as follows.

Claim 3, lines 1-2, replace "either of the previous claims" with --Claim 1--.

Claim 4, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 6, line 1, delete "or Claim 5".

Claim 7, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 8, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 10, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 11, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 12, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

Claim 14, line 1, replace "one of Claims 1 to 11" with --Claim 1--.

Claim 15, lines 1-2, replace "one of the preceding claims" with --Claim 1--.

REMARKS

Claims 1-15 are active in this application.

The claims have been amended to remove multiple dependencies. No new matter is believed to have been added to this application by these amendments.

Applicant submits that the present application is ready for examination on the merits.

Early notice to this effect is earnestly solicited.

Respectfully submitted,

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GLAZING HAVING ELECTRICALLY CONTROLLABLE
OPTICAL AND/OR ENERGY PROPERTIES

5 The present invention relates to glazing having
electrically controllable optical and/or energy
properties.

10 Thus, it relates to glazing some of whose
characteristics can be modified by a suitable
electrical supply, most particularly the transmission,
absorption and reflection within certain wavelengths of
electromagnetic radiation, especially in the visible
and/or in the infrared, or else the light scattering.

15 There is in fact an increasing demand for so-
called "smart" glazing whose properties may be varied.

20 Thus, from the thermal standpoint, glazing
whose transmission/absorption may be varied within at
least part of the solar spectrum allows the solar heat
influx into rooms or passenger areas/compartments to be
controlled when it is fitted as the external glazing in
buildings or as windows in transportation means of the
type comprising cars, trains, aeroplanes, etc., and
thus it allows excessive heating of the latter to be
prevented should there be strong sunlight.

25 From the optical standpoint, the glazing allows
the degree of vision to be controlled, thereby making
it possible to prevent glare should there be strong
sunlight, when it is mounted as exterior glazing. It
may also have a particularly advantageous shutter
effect, both as exterior glazing and if it is used as
interior glazing, for example for equipping internal
partitions between rooms (offices in a building), or
for isolating compartments in trains or aeroplanes, for
example.

35 There are many other applications: for example,
glazing having variable light transmission/reflection
may be used for making rear-view mirrors, which can
darken as required in order to prevent the driver of
the car becoming dazzled. They may also be used for
indicating panels on roadways, or any display panel,

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for example so as to reveal the drawing/message only intermittently in order to attract greater attention.

One particularly advantageous application of the systems having variable light absorption relates to display screens, especially all those with which televisions and computing hardware are equipped. This is because this type of glazing makes it possible to improve the contrast of the image, especially taking the ambient brightness into account.

The advantage that such glazing may provide justifies the fact that many systems have already been studied.

Thus, known systems allowing the light transmission or absorption of glazing to be varied are especially so-called viologen-based systems, such as those described in Patent US-5,239,406 or in Patent EP-A-0,612,826. These make it possible to obtain variable absorption, essentially in the visible region.

To the same end, there are also so-called electrochromic systems, the operating principle of which will be briefly recalled: these comprise, in a known manner, a layer of an electrochromic material capable of reversibly and simultaneously inserting ions and electrons, the oxidation states of which electrochromic material corresponding to the inserted and extracted states have a distinct colour, one of the states having a higher light transmission than the other. The insertion or extraction reaction is controlled by a suitable electrical supply using a current generator or a voltage generator. The electrochromic material, usually based on tungsten oxide, must thus be brought into contact with a source of electrons, such as a transparent electrically conductive layer, and with a source of ions (cations) such as an ionically conductive electrolyte.

Moreover, it is known that, in order to guarantee at least the order of a hundred switching operations, the layer of electrochromic material must be connected to a counterelectrode which is itself

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5 The counterelectrode must consist of a layer which is either neutral in terms of colour or is at least transparent or barely coloured when the electrochromic layer is in the bleached state. Since tungsten oxide is a cathodic electrochromic material, 0 that is to say its coloured state corresponds to the most reduced state, an anodic electrochromic material based on nickel oxide or iridium oxide is generally used for the counterelectrode. It has also been proposed to use an optically neutral material in the 5 oxidation states in question, such as, for example, cerium oxide or organic materials such as electronically conductive polymers (polyaniline, etc.) or Prussian blue.

At the present time, these systems may be put into two categories, depending on the type of electrolyte that they use:

Other systems use electrochromic-type reversible ion-insertion materials slightly

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differently. These are, for example, so-called
gasochromic systems, in which the electrochromic
material is provided with a thin catalytic layer
capable of decomposing hydrogen and mounted in a
5 double-glazing unit on the internal gas-cavity side: by
sending hydrogen into the internal space of the double-
glazing unit, tungsten oxide becomes coloured. It
returns to the bleached state by injecting oxygen,
instead of hydrogen, into the internal space.

10 These systems having one or more reversible-
insertion materials are particularly advantageous in
the sense that they allow the absorption to be varied
over a broader wavelength range than viologen-based
systems: they allow variable absorption not only in the
15 visible but also, in particular, in the infrared, which
may confer on them an effective optical and/or thermal
role.

 Viologen-based or electrochromic systems,
deposited on or associated with transparent substrates,
20 form glazing whose light absorption and transmission
(as well as energy transmission) may vary within given
ranges, especially ranges determined by the choice of
electrochromic materials used and/or by the choice of
their thicknesses.

25 Another type of "smart" glazing is formed by
what is termed an "optical valve": this is a film
comprising a generally crosslinked polymer matrix in
which microdroplets are dispersed, these microdroplets
containing particles which have the property of being
30 aligned in a preferred direction due to the action of
an electric or magnetic field. The film has variable
optical properties depending in particular on the
potential applied to the terminals of the conductive
layers placed on either side of the film and on the
35 concentration and nature of the orientable particles.
Thus, Patent WO-93/09460 discloses an optical valve
based on a film comprising a crosslinkable poly-
organosilane matrix and inorganic or organic orientable
particles, more particularly light-absorbing particles

such as particles of polyiodides. When a voltage is applied to the film, the particles intercept the light much less than when no voltage is applied.

Glazing with variable light scattering, the operating principle of which is similar, is also known by the expression "liquid-crystal glazing". This is based on the use of a film placed between two conductive layers based on a polymeric material in which droplets of liquid crystals are dispersed, especially nematic liquid crystals of positive dielectric anisotropy. When a voltage is applied to the film, the liquid crystals orient in a preferred direction, thereby allowing vision. When no voltage is applied, and the crystals are not aligned, the film becomes scattering and prevents vision. Examples of such films are described, for instance, in European Patent EP-0,238,164 and United States Patents US-4,435,047, US-4,806,922 and US-4,732,456. This type of film, once laminated and incorporated between two glass substrates, is sold by Saint-Gobain Vitrage under the brand name "Priva-lite". In fact, it is possible to use any of the liquid-crystal devices known by the term "NCAP" (Nematic Curvilinearly Aligned Phase) or the term "PDLC" (Polymer Dispersed Liquid Crystal) or the term "CLC" (Cholesteric Liquid Crystal). These may furthermore contain dichroic dyes, especially in solution in the liquid-crystal droplets. It is then possible to jointly vary the light scattering and the light absorption of the systems.

It is also possible to use, for example, gels based on cholesteric liquid crystals containing a small amount of crosslinked polymer, such as those described in Patent WO-92/19695.

However, all these various systems/glazing assemblies have limits which are intrinsic to them, which limits relate especially to their optical appearance.

Thus, in the case of electrochromic-type glazing, it is possible to vary the range of accessible

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values of light transmission (T_L) by adjusting, for example, the thickness of the layer (or layers) based on a reversible-insertion material. However, for a given system, the T_L range can only be shifted somewhat towards lower or higher values, and this cannot be easily extended. Furthermore, the choice of reversible-insertion material will have a bearing on the colorimetric appearance of the glazing both in transmission and in reflection.

The object of the invention is therefore to alleviate these drawbacks, especially by proposing novel electrically controllable glazing having variable optical and/or energy properties, the optical appearance of which can also be varied.

The subject of the invention is glazing which incorporates at least one electrically controllable system having variable optical and/or energy properties, of the type comprising a system having variable light and/or energy transmission/absorption and/or variable light scattering. This glazing furthermore includes at least one means for adjusting the optical appearance conferred on the glazing by the electrically controllable system. This means is advantageously in the form of at least one coating having antireflection properties in the visible or in the near infrared.

In order to simplify matters, hereafter this coating will be referred to as an "antireflection coating" and the electrically controllable system will be referred to as the "functional system".

Combining the functional system with an antireflection coating, the characteristics of which may be adjusted precisely, makes it actually possible to modify the optical or thermal performance of the glazing. Thus, the antireflection coating may have an influence on the range of light or solar transmission that the glazing may have with an *ad hoc* electrical supply: in particular, it may shift, in a controlled manner, this range towards higher T_L or T_E (energy

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transmission) values. Specifically, this means that, for a given functional system, the addition of the antireflection coating will make it possible to modify its T_L or T_E range depending on the intended application, without having to modify the functional system itself. With regard to the manufacture of the glazing, this allows production which is much more flexible and rational than if as many different functional systems as there are envisaged applications had to be manufactured.

This is because, for some applications, the aim is to provide a strongly colouring/absorbing effect when a voltage is applied, making accommodations for a certain residual coloration when no voltage is applied (for example if it is desired to tend towards glazing having a shutter effect in the coloured state).

On the other hand, for other applications, it will be necessary for the glazing to have, when no voltage is applied, little or even no residual coloration. This may be the case, for example, in functional systems for display screens. Furthermore, it is much simpler to maintain only as restricted a number of "standard" functional systems as possible and to adapt them by means of the *ad hoc* antireflection coating, which coating is generally much less complicated to manufacture than the functional systems.

The antireflection coating may also allow the range of accessible T_L or T_E values to be extended. This is a very important advantage whatever the application intended, which advantage is particularly essential when the glazing is used to enhance the contrast of display screens. This is most particularly true with new televisions making use of flat-screen, plasma technology, these also being called emissive, which tend to have a much lower brightness than standard cathode-ray-tube televisions.

One advantageous embodiment of the antireflection coating consists in depositing it on at least one of the external faces of the glazing, that is

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to say the faces exposed directly to the ambient atmosphere of the glazing. Both these faces may be treated or, in the case of display-screen glazing, only the face turned towards the outside of the apparatus.

- 5 In a known manner, this coating may comprise a stack of thin layers having alternately high and low refractive indices, which, by an interference effect, tend to reduce the light reflection of the glazing to the profit of an increase in its light transmission.
- 10 Examples of antireflection stacks are, for example, known from Patents EP-0,728,712, EP-0,712,815 and EP-0,791,562.

These layers are generally made of a dielectric material of the oxide type (SiO_2 or Al_2O_3 for the layers

15 having a low index of less than 1.7 and SnO_2 , TiO_2 or Nb_2O_5 for the layers having a higher index of at least 1.9) or else of the fluoride type (MgF_2 as the low-index layer) or of the nitride type, such as Si_3N_4 or silicon derivatives of the SiO_xN_z or SiO_xC_y type.

- 20 However, an antistatic function may also be conferred on the antireflection coating by incorporating into the coating, as a low-index or high-index layer, a layer of a material which, from an electrical standpoint, is at least slightly conductive.
- 25 A layer of a doped metal oxide may especially be chosen, such as F:SnO_2 or ITO (tin-doped indium oxide), which has an index of at least 1.9 to 2.0 or a layer of a conductive polymer.

The antireflection coating may also consist

30 only of a single layer having a refractive index gradient in its thickness, this layer being obtained, for example, using a pyrolysis-type deposition technique. This hot deposition technique makes it possible to obtain layers which are particularly

35 durable from a mechanical standpoint, which may be very important depending on the envisaged application of the system, especially so that the coating can withstand being touched, being repeatedly cleaned, etc. This graded-index layer has in fact a chemical composition

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which varies through its thickness, for example gradually approaching a SiO_2 -type composition from one of the SiOC or SiON type.

5 The glazing according to the invention may also include a means for adjusting the optical appearance conferred on the said glazing by the functional system, comprising at least one coating for attenuating/modifying the colour of the glazing in reflection (this coating being as an addition or an alternative to the
10 previous antireflection coating). This is because, in some applications, for example in the case of glazing for display screens of all kinds, it is preferable for the colour of the glazing in reflection to be as neutral as possible, especially so that in the
15 completely bleached state the glazing does not appear tinted at all and so that it is of a tint modifying the colour of the image appearing on the screen as little as possible. Since the antireflection coating makes it possible for the intensity of light reflection of the
20 glazing to be generally lowered, this other coating may therefore complete its optical role by allowing its colour in reflection to be attenuated, in practice by lowering the C^* saturation values, in the (L, a^*, b^*) colorimetry system, of the glazing in reflection.

25 This coating is advantageously in contact with the functional system, in the form of a thin layer at least having a refractive index intermediate between those of the materials with which it is in contact on each of its faces. This may especially be a thin layer
30 having a refractive index of between 1.6 and 1.9, especially one based on aluminium oxide Al_2O_3 , on aluminium oxynitride AlN or on yttrium oxide Y_2O_3 , on silicon oxycarbide and/or oxynitride SiOC , SiON , or on a mixture of at least two of these materials, which may
35 be deposited by vacuum technologies of the sputtering type or by pyrolysis-type technologies, the latter being most particularly indicated for depositing layers of silicon derivatives.

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This coating may comprise not one layer, but several, especially in the form of at least two superposed layers whose average index is, for example, between 1.6 and 1.9, for example an $\text{SnO}_2/\text{SiO}_2$ or
5 $\text{SnO}_2/\text{SiO}_2/\text{SnO}_2$ stack.

This coating may also be a layer having an index gradient through its thickness so as to optimize its adjustment with respect to the indices of the materials which surround it. The formation of such a
10 graded-index layer using a vapour-phase pyrolysis technique (also called CVD for "Chemical Vapour Deposition") is, for example, described in Patent WO-97/03029 (to which reference may also be made for the graded-index layer of the previous antireflection
15 coating).

The glazing according to the invention may also include a primer/tie-layer coating for the functional system with respect to its carrier substrate, which may prove to be particularly advantageous if the nature of
20 the substrate is polymeric/plastic and not mineral, of the glass type. The coating may comprise a thin metal layer, a layer of a silicon derivative of the SiO_2 type or of a suitable metal oxide of the Al_2O_3 type. It may also be a tie-layer varnish. Advantageously, this tie-
25 layer coating may also be made to fulfil a role of attenuating the colour of the glazing in reflection, like the specific coating mentioned above, especially if it has a refractive index matching that of the plastic substrate and that of the layer of the
30 functional system with which it is in contact.

The glazing according to the invention may also include a hydrophilic coating having antimisting properties or a hydrophobic coating having anti-rain properties on at least one of its external faces. As
35 suitable hydrophobic coating, reference may be made, for example, to Patents EP-799,873 and EP-692,463. It may especially be at least one layer consisting of a composition having at least one fluoroalkoxysilane, the alkoxy functional groups of which are directly linked

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to the silicon atom, a system of one or more aqueous solvents and at least one catalyst chosen from an acid and/or a Brönsted base. The coating may also include a primer layer promoting adhesion of the hydrophobic layer to the glass, for example a primer layer based on silanes.

The glazing according to the invention may also include a coating having photocatalytic properties giving the glazing antifouling properties, especially on at least one of its external faces. This may especially be a coating comprising semiconductor materials, of the crystallized oxide or sulphide type, having this type of property, especially crystallized oxides of the ZnO, WO₃ or SnO₂ type and more particularly titanium oxide at least partially crystallized in anatase form. This type of coating and the various ways of obtaining it are especially described in Patents WO-97/10186 and WO-97/10185. These coatings make it possible to degrade any dirt of organic nature. They may furthermore be hydrophilic and thus promote the removal of inorganic dirt as well.

The glazing according to the invention may also include at least one coating having electromagnetic screening properties, especially screening properties with respect to radiation emitted by emissive screens of the plasma-screen type. This type of coating includes, for example, at least one thin layer essentially made of metal, or of a conductive metal oxide, and/or one or more superposed arrays of metal conducting wires and/or a metal mesh.

The functional system of the glazing is, as was seen previously, generally in the form of a superposition of functional layers placed between two carrier/protective substrates which may be rigid, semi-rigid or flexible. It may be advantageous to use, as carrier substrate, at least one of the rigid substrates of which the glazing is composed and/or at least one flexible carrier substrate which may be associated, by lamination, with one of the rigid substrates of which

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the glazing is composed. The functional system may also be placed on a suitable substrate, and then simply protected/encapsulated by an impermeable protective coating providing it with a degree of mechanical protection. It may be an inorganic layer of the SiO_2 or Si_3N_4 type. It may also be of the polymer(s) type instead, especially in the form of a varnish (epoxy or polyparaxylylene) or of a lacquer (polyurethane or polyacrylic). Preferably, it may be a vacuum-deposited monolayer of a polymer.

The object of the invention is also the use of the glazing described above as glazing for buildings, especially as exterior windows, windows for interior partitions or glazed doors (for example sunroofs of the "Vélux" type), and as glazing with which transportation means are equipped, especially windows for motor vehicles (sunroofs and front and rear side windows) railway windows or aeroplane windows, especially as windscreens, windscreen top-tint strips, or cabin windows. Such glazing is also indicated for equipping display screens in order to enhance image contrast, especially television or computer screens. Such glazing may also be used as protection for solar panels (satellites), for camera lenses, for spectacles for aircraft pilots, sunglasses or ophthalmic spectacles, or else as glazing suitable for the protection of objects/plants from heat or strong light which are sensitive thereto, for example for equipping greenhouses or shop windows.

As mentioned above, the invention thus applies to various types of electrochemically controllable glazing. This may be, as mentioned, glazing having variable light transmission/absorption, especially having a viologen-based or electrochromic system, particularly of the type of those described in the aforementioned Patents EP-0,338,876, EP-0,408,427, EP-0,575,203 and EP-0,628,849. It is preferably in the form of a stack of functional layers comprising, in succession, a preferably transparent electrically

conductive layer, a so-called cathodic electrochromic layer capable of reversibly inserting cations such as H^+ , Li^+ , Na^+ , Ag^+ , a layer of electrolyte, optionally a counterelectrode in the form of a second, so-called
5 anodic, electrochromic layer which is also capable of reversibly inserting cations, and finally a second electrically conductive layer.

With regard to the nature of the electrically conductive layers of the device, there are two possible
10 variants. It is possible to use materials based on doped metal oxides, such as fluorine-doped tin oxide $F:SnO_2$ or tin-doped indium oxide ITO. It is also possible to use layers of metals or of metal alloys, for example based on gold Au, silver Ag or aluminium
15 Al. Since the device generally possesses two electrically conductive layers, they may both be metal layers, or they may both be based on doped oxides, or one may be based on metals and the other based on doped oxides. It is also possible to superpose several
20 electrically conductive layers of different types, for example a doped-oxide layer associated with at least one metal layer in a stack of the ITO/Ag or Au/ITO type, for example.

These layers (or at least one of them) may also
25 be made of one or more conductive polymers.

In order to form the layer of cathodic electrochromic material, a material, or a mixture of materials, chosen from the group comprising tungsten oxide WO_3 , molybdenum oxide MoO_3 , vanadium oxide V_2O_5 ,
30 niobium oxide Nb_2O_5 , titanium oxide TiO_2 , a "cermet" material (a combination of metallic and ceramic material, especially in the form of metal particles in a ceramic matrix) such as WO_3/Au or WO_3/Ag , and a mixture of tungsten and rhenium oxides WO_3/ReO_3 , may be
35 chosen. These materials are especially suitable in the case of the reversible insertion of lithium ions. If the device operates by the reversible insertion of protons, the same materials may be used, but this time in hydrated form.

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In order to form the layer of anodic electrochromic material, a material which satisfies the formula $M_xA_yU_z$, where M is a transition metal, A is the ion used for the reversible insertion, for example an alkali metal or a proton, and U is a chalcogen, such as oxygen or sulphur, may be chosen.

It may, especially in the case of the insertion of proton ions H^+ , be a compound or a mixture of compounds belonging to the group comprising $LiNiO_x$, IrO_xH_y , $IrO_xH_yN_z$, NiO_x , $NiO_xH_yN_z$, RhO_x , CoO_x , MnO_x and RuO_x . In the case of the reversible insertion of lithium ions Li^+ , a compound or a mixture of compounds belonging to the group comprising $LiNiO_x$, $LiMn_2O_4$, $Li_xS_nO_y$, IrO_x , Li_xIrO_y , $Li_xS_nO_y$, NiO_x , CeO_x , TiO_x , CeO_x-TiO_x , RhO_x , CoO_x , CrO_x and MnO_x is chosen instead.

With regard to the choice of electrolyte material, there are in fact two types of this, as was mentioned above.

It may be a layer of aqueous liquid, such as water to which sulphuric or phosphoric acid has been added, in the case of the reversible insertion of protons and may be a layer of anhydrous liquid, such as propylene carbonate containing a lithium salt, in the case of the reversible insertion of lithium ions. It may also be a layer of gel or of polymer, especially protonically conductive polymers of the type comprising a solid solution of polyoxyethylene and of phosphoric acid POE- H_3PO_4 .

However, it may also be an electrolyte in the form of a solid material, especially one based on a metal oxide. According to a variant of the invention, the system is chosen so that it contains only layers of solid materials. Within the context of the invention, the term "solid material" should be understood to mean any material having the mechanical integrity of a solid, particularly any essentially inorganic or organic material or any hybrid material, that is to say one that is partially inorganic and partially organic, such as materials that may be obtained by sol-gel

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deposition from organo-inorganic precursors. The configuration is then one of a so-called "all-solid" system which is an advantage in terms of ease of manufacture. This is because, when the system contains
5 an electrolyte in the form of a polymer which does not have, for example, the mechanical integrity of a solid, this requires in fact the manufacture, in parallel, of two "half-cells" each consisting of a carrier substrate coated with a first electrically conductive layer and
10 then with a second, electrochemically active layer, these two half-cells then being joined together with the electrolyte inserted between them. With an "all-solid" configuration, the manufacture is simplified since all the layers of the system may be
15 deposited, one after the other, on a single carrier substrate. Thus all the operations for manufacturing the glazing are simplified since there is now only a single substrate intended to receive layers instead of two.

20 Furthermore, whether the electrolyte is a "solid" electrolyte or not, it may comprise a layer made of an ionically conductive material capable of reversibly inserting the ions, but the degree of oxidation of which is maintained essentially constant.
25 It may especially be a material having electrochromic properties, as described in the aforementioned Patent EP-97/400702.3.

The functional system of the element according to the invention may therefore be placed either between
30 two substrates, or on a single substrate, more particularly in the case of an "all-solid" system. The rigid carrier substrates are preferably made of glass, acrylic polymer, polycarbonate or certain polyurethanes. The carrier substrates may also be soft,
35 flexible and intended to be laminated to rigid substrates; it may be a soft polycarbonate, a polyethylene terephthalate (PET), etc. The lamination may be carried out with intercalated sheets of polymer of the thermoplastic type, such as polyvinyl butyral

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(PVB), ethylene-vinyl acetate (EVA) or certain polyurethanes. With no lamination carried out, the system may also be provided with a protective varnish or film, as mentioned above.

5 Such glazing may thus have a "monolithic" structure, that is to say comprises a single rigid substrate, or a plurality of rigid substrates, a laminated and/or multiple-glazing structure, or else a
10 plastic layer, especially based on polyurethane, which structure is especially described in Patents EP-191,666, EP-190,953, EP-241,337, EP-344,045, EP-402,212, EP-430,769 and EP-676,757.

15 By way of example, the glazing according to the invention may thus have a structure or sequence of the type:

20 antireflection coating/glass 1/coating
attenuating or modifying the colour in reflection/
functional system/intercalated sheet of polymer of the
PU type/glass 2

25 This system may also be combined with another glass in order to form a double-glazing unit. It is also possible to provide an intercalated sheet of polymer of the PU type and another glass between the
30 glass 1 and the coating attenuating the colour in reflection: it is thus possible to appose one structure (antireflection coating/glass 1) with another structure (functional system/coating attenuating the colour/glass), these structures being laminated with a sheet of polymer.

 Further details and advantageous characteristics of the invention emerge from the description given below of various non-limiting embodiments, with reference to the appended drawing which shows:

35 ☐ **Figure 1:** electrochromic glazing having a laminated structure in cross section

 This figure is extremely schematic and does not respect the proportions between the various elements shown, so as to make it easier to understand. In

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particular, all the electrical connections, which are known per se, are not shown.

The rigid substrates used for all the following examples are substrates made of silicon-soda-lime glass 4 mm in thickness (their thickness may in fact be chosen especially within the 0.7 to 6 mm range).

These are so-called "clear" glass substrates sold by Saint-Gobain Vitrage under the name Planilux.

EXAMPLE 1

Figure 1 shows electrochromic glazing having a laminated structure comprising two glass plates in a configuration suitable, for example, to be used as the display screen of a flat-screen television: it shows two clear glass plates 1 and an electrochromic functional system 3 of the "all-solid" type, consisting of the stack of the following functional layers and a sheet of polyurethane 13:

□ a first, electrically conductive layer 4 made of F:SnO₂ 500 nm in thickness;

□ a first layer 5 of anodic electrochromic material made of iridium oxide IrO_x (hydrated) 30 nm in thickness (it could be replaced with a layer of hydrated nickel oxide);

□ a layer 6 of hydrated tantalum oxide Ta₂O₅.H_x 5 nm in thickness, having a tie-layer function;

□ a layer 7 of tungsten oxide 200 nm in thickness;

□ a second layer 8 of hydrated tantalum oxide 200 nm in thickness;

□ a second layer 9 of cathodic electrochromic material based on tungsten oxide H_xWO₃ 380 nm in thickness;

□ a second layer 10 of ITO 280 nm in thickness.

Between the electrically conductive layer 4 and the glass 2 there is a coating 11 whose function is to attenuate the colour of the glazing in reflection: it is a layer of silicon oxycarbide SiOC having an index of approximately 1.7 and a geometrical thickness of approximately 50 to 55 nm (deposited in a known manner

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by CVD on the glass 2). Its index is thus intermediate between those of the materials which surround it, namely that of the glass 1 (approximately 1.5) and that of the layer 4 of F:SnO₂ (approximately 2).

5 Deposited on the external face of the glass 2 is an antireflection coating 12 composed of the succession of the following layers (starting from the surface of the glass 2):

SnO₂(19 nm)/SiO₂(33 nm)/Nb₂O₅(115 nm)/SiO₂(88 nm)

10 The coating was deposited in a known manner on the glass 2 by magnetic-field-assisted reactive sputtering in the presence of oxygen using suitable metal/silicon targets.

15 The coating 12/glass 2/coating 11/functional system 3 assembly is then laminated to the glass 1 by means of a sheet 13 of organic polymer of the polyurethane type having a thickness of at least 1.24 mm.

20 The glazing is mounted so that the glass 2 is the glass facing the outside of the screen.

25 It appears that by combining the two types of optical coatings 11 and 12 with the functional system 3, it is possible to shift the accessible T_L range to higher values and to reduce the intensity of the residual colour, in reflection, in the bleached state and in the coloured state.

EXAMPLE 2

30 An Example 2 was produced using the same coatings 11 and 12 and the same functional system 3 on the same glass 2. Thereafter, the only difference is the way in which the glass is mounted: in this case, the final ITO layer 10 of the functional system has been simply surmounted by a vacuum-deposited layer of polyparaxylylene varnish. This thus results in a
35 single-glass structure of the type: coating 12/glass 1/coating 11/functional system 3/varnish.

EXAMPLE 3

 An Example 3 was produced in a manner similar to Example 1. Only some of the thicknesses of the

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layers of the electrochromic system differ slightly. Furthermore, the layer 6 of hydrated tantalum oxide was omitted. The stack of the electrochromic system in this Example 3 is therefore as follows:

- 5 □ a first, electrically conductive layer 4' of F:SnO₂ 500 nm thickness;
- a first layer 5' of anodic electrochromic material made of iridium oxide IrO_x (hydrated) 37 nm in thickness (it could be replaced with a layer of
- 10 hydrated nickel oxide);
- a layer 7' of tungsten oxide 100 nm in thickness;
- a second layer 8' of hydrated tantalum oxide 100 nm in thickness;
- 15 □ a second layer 9' of cathodic electrochromic material based on tungsten oxide H_xWO₃ 280 nm in thickness;
- a second layer 10' of ITO 270 nm in thickness.

20 On the other hand, in the case of the coating 11 and the coating 12 the mounting of the electrochromic system is identical to that in Example 1.

EXAMPLE 4

25 An Example 4 was produced like Example 3, apart from the fact that, in this example, there is no antireflection coating 12.

 It was confirmed that the optical properties of the glazing were improved when at least one coating

30 (coating 11) attenuating the colour or an antireflection coating (coating 12) was provided, the maximum improvement was obtained by using both types of coating together. It is possible to provide a second antireflection coating (so that each of the external

35 faces of the external substrates 1 and 2 is treated).

 The following optical properties of Examples 3 and 4 in the bleached state (+1.2 V supply) and in the coloured state (-1.6 V supply) were compared:

 / the light transmission T_L (%);

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- 10

Table 2 below gives data on the energy properties of these same two examples, namely the energy transmission $T_E(\%)$, the energy reflection R_{E1} (on the external side) and R_{E2} (on the internal side).

Also measured were their solar factors SF (the solar factor is the ratio between the total energy entering the room through the glazing to the incident solar energy):

- 5 ➤ for Example 3: the SF is 33% in the coloured state (-1.6 V) and 73% in the bleached state (+1.2 V);
- for Example 4: the SF is 32% in the coloured state and 67% in the bleached state.

10 It may be seen from this data that, in the case of Example 3 according to the invention, it is possible to achieve a wider light transmission range and, in particular, to achieve a T_L of almost 80% in the bleached state. The energy transmission in the bleached state of Example 3 is also lower than that of Example 4
15 and the energy reflections are higher, whether in the coloured state or in the bleached state. Example 4, which has only the anti-colour coating, already shows an improvement over standard electrochromic glazing, especially with regard to R_{L1} and R_{L2} colorimetry in
20 reflection. But Example 3, in which an antireflection coating has been added, allows the T_L range to be broadened towards higher values and allows the glazing to be made more effective from the standpoint of the filtration of thermal, especially solar, radiation.

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CLAIMS

1. Glazing which incorporates at least one electrically controllable system having variable optical and/or energy properties, especially in the form of a system comprising one or more reversible-insertion materials of the electrochromic-system (3) or gasochromic-system type, in the form of an optical-valve or viologen-based system or in the form of a liquid-crystal or cholesteric-gel system, **characterized in that** it also includes at least one means for adjusting the optical appearance conferred on the said glazing by the said system, these means comprising at least one coating (12) having antireflection properties in the visible.

2. Glazing according to Claim 1, **characterized in that** the coating (12) having antireflection properties is deposited on at least one of its external faces and comprises a stack of thin layers having alternately high and low refractive indices or a graded-refractive-index layer.

3. Glazing according to either of the previous claims, **characterized in that** the coating (12) having antireflection properties also has antistatic properties, by including a stack of thin layers at least one of which is made of an electrically conductive material of the doped-metal-oxide or conductive-polymer type.

4. Glazing according to one of the preceding claims, **characterized in that** it also includes a means for adjusting the optical appearance conferred on the said glazing by the said system, comprising at least one coating (11) for attenuating/modifying the colour of the glazing in reflection.

5. Glazing according to Claim 4, **characterized in that** the coating (11) for attenuating/modifying the colour of the glazing in reflection is in contact with the electrically controllable system, in the form of a thin layer having a refractive index intermediate

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between those of the materials with which it is in contact on each of its faces.

6. Glazing according to Claim 4 or Claim 5, **characterized in that** the coating (11) for
5 attenuating/modifying the colour of the glazing in reflection includes a thin layer having a refractive index of between 1.6 and 1.9, especially one based on aluminium oxide Al_2O_3 , on yttrium oxide Y_2O_3 , on silicon oxycarbide and/or oxynitride SiOC , SiON , or on a
10 mixture of at least two of these materials, or at least two superposed thin layers whose average refractive index is between 1.6 and 1.9, especially an $\text{SnO}_2/\text{SiO}_2$ or $\text{SnO}_2/\text{SiO}_2/\text{SnO}_2$ stack.

7. Glazing according to one of the preceding
15 claims, **characterized in that** it includes a primer/tie-layer coating for the electrically controllable system (3) with respect to its carrier substrate (2), especially when the latter is a polymeric/plastic material.

20 8. Glazing according to one of the preceding claims, **characterized in that** it also includes a coating having hydrophilic/antimisting properties or having hydrophobic/anti-rain properties on at least one of its external faces.

25 9. Glazing according to Claim 8, **characterized in that** the coating having hydrophobic properties includes at least one layer consisting of a composition having at least one fluoroalkoxysilane, the alkoxy functional groups of which are directly linked to the silicon
30 atom, a system of one or more aqueous solvents and at least one catalyst chosen from an acid and/or a Bronsted base.

10. Glazing according to one of the preceding
35 claims, **characterized in that** it also includes a coating having photocatalytic/antifouling properties, comprising especially TiO_2 at least partially crystallized in the anatase form, especially on at least one of its external faces.

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11. Glazing according to one of the preceding claims, **characterized in that** it also includes at least one coating having electromagnetic screening properties.
- 5 12. Glazing according to one of the preceding claims, **characterized in that** the electrically controllable system (3) is a superposition of functional layers placed between two carrier substrates (1, 2), each of the said substrates possibly being
- 10 rigid, semi-rigid or flexible.
13. Glazing according to Claim 12, **characterized in that** the electrically controllable system (3) uses, as carrier substrate, at least one of the rigid substrates (2) of which the glazing is composed, and/or at least
- 15 one flexible carrier substrate (13) associated, by lamination, with one of the rigid substrates (1) of which the said glazing is composed.
14. Glazing according to one of Claims 1 to 11, **characterized in that** the electrically controllable
- 20 system (3) is a superposition of functional layers placed on a carrier substrate (2) and provided with a protective film of the inorganic or polymeric layer type, especially in the form of a lacquer or of a varnish.
- 25 15. Use of the glazing according to one of the preceding claims as glazing for buildings, especially an exterior window or a window for an internal partition or glazed door, as glazing with which the internal partitions or windows of transportation means
- 30 of the train, plane, car or boat type are equipped, as glazing for a display screen of the computer- or television-screen type, for spectacles or camera lenses or as protection for solar panels.

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ABSTRACT

The subject of the invention is glazing which incorporates at least one electrically controllable system having variable optical and/or energy properties, especially in the form of a system comprising one or more reversible-insertion materials of the electrochromic-system type, in the form of an optical-valve or viologen-based system or in the form of a liquid-crystal or cholesteric-gel system. This glazing also includes at least one means for adjusting the optical appearance conferred on the said glazing by the said system, these means comprising at least one coating having antireflection properties in the visible.

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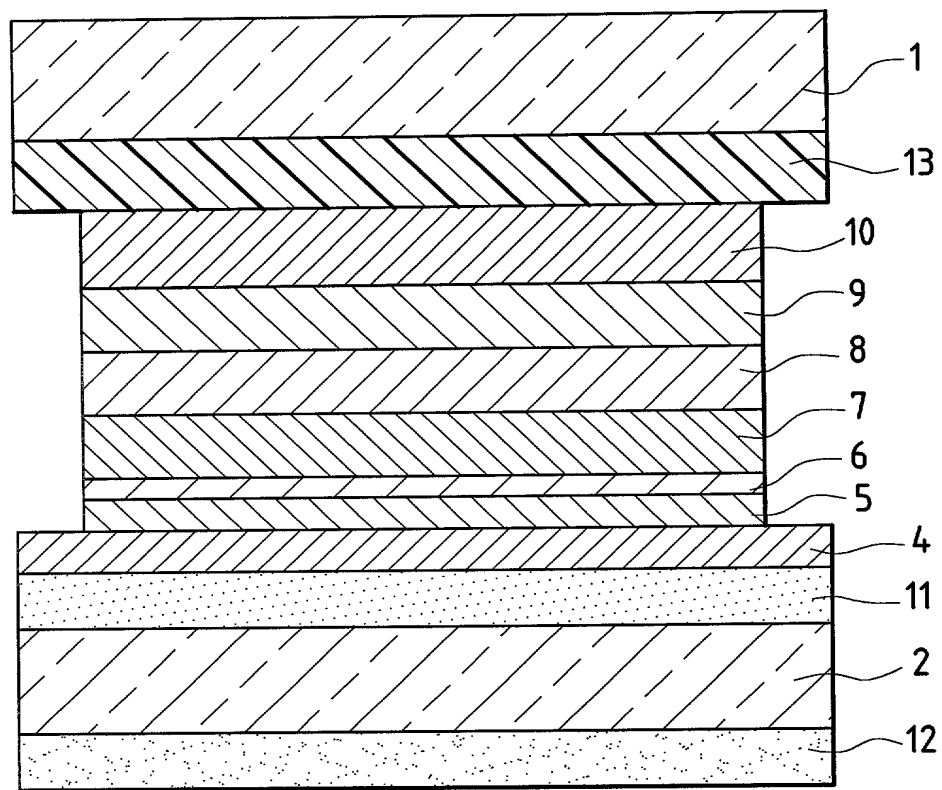


FIG.1

Declaration and Power of Attorney for Patent Application

Déclaration et Pouvoirs pour Demande de Brevet

French Language Declaration

En tant l'inventeur nommé ci-après, je déclare par le présent acte que.

Mon domicile, mon adresse postale et ma nationalité sont ceux figurant ci-dessous à côté de mon nom

Je crois être le premier inventeur original et unique (si un seul nom est mentionné ci-dessous), ou l'un des premiers co-inventeurs originaux (si plusieurs noms sont mentionnés ci-dessous) de l'objet revendiqué, pour lequel une demande de brevet a été déposée concernant l'invention intitulée

et dont la description est fournie ci-joint à moins

☐ ci-joint

☐ a été déposée le _____

sous le numéro de demande des Etats-Unis ou le
numéro de demande international PCT

_____ et modifiée le

_____ (le cas échéant)

Je déclare par le présent acte avoir passé en revue et compris le contenu de la description ci-dessus, revendications comprises, telles que modifiées par toute modification dont il aura été fait référence ci-dessus

Je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

GLAZING WITH OPTICAL AND/OR ENERGETIC

PROPERTIES CAPABLE OF BEING ELECTRICALLY

CONTROLLED (as amended)

the specification of which

☐ is attached hereto.

☒ was filed on March 8, 2000

as United States Application Number or PCT
International Application Number

09/486,719 and was amended on

_____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119(a)-(d) ou § 365(b) du Code des Etats-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur ou, en vertu du Titre 35, § 365(a) du même Code, sur toute demande internationale PCT désignant au moins un pays autre que les Etats-Unis et figurant ci-dessous et, en cochant la case, j'ai aussi indiqué ci-dessous toute demande étrangère de brevet, tout certificat d'inventeur ou toute demande internationale PCT ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

Prior foreign application(s)

Demande(s) de brevet antérieure(s)

98/08808	FRANCE
(Number)	(Country)
(Numéro)	(Pays)
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(Number)	(Country)
(Numéro)	(Pays)

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(Application No.)	(Filing Date)
(N° de demande)	(Date de dépôt)
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(Application No.)	(Filing Date)
(N° de demande)	(Date de dépôt)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des Etats-Unis, de toute demande de brevet effectuée aux Etats-Unis, ou en vertu du Titre 35, § 365(c) du même Code, de toute demande internationale PCT désignant les Etats-Unis et figurant ci-dessous et, dans la mesure où l'objet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande antérieure américaine ou internationale PCT, en vertu des dispositions du premier paragraphe du Titre 35, § 112 du Code des Etats-Unis, je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la demande antérieure et la date de dépôt de la demande nationale ou internationale PCT de la présente demande:

PCT/FR99/01652	08 July 1999
(Application No.)	(Filing Date)
(N° de demande)	(Date de dépôt)
<hr/>	
(Application No.)	(Filing Date)
(N° de demande)	(Date de dépôt)

Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d'une incarcération, ou des deux, en vertu de la Section 1001 du Titre 18 du Code des Etats-Unis, et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby claim foreign priority under Title 35, United States Code, § 119(a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below, by checking the box, and have also identified below any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Not Claimed
Droit de priorité non revendiqué

09 July 1998	<input type="checkbox"/>
(Day/Month/Year Filed)	
(Jour/Mois/Année de dépôt)	
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(Day/Month/Year Filed)	<input type="checkbox"/>
(Jour/Mois/Année de dépôt)	

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below.

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(Status) (patented, pending, abandoned)
(Statut) (breveté, en cours d'examen, abandonné)

(Status) (patented, pending, abandoned)
(Statut) (breveté, en cours d'examen, abandonné)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

French Language Declaration

POUVOIRS. En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) et/ou agent(s) suivant(s) pour qu'ils poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire s'y rapportant avec l'Office des brevets et des marques (mentionner le nom et le numéro d'enregistrement).

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)

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(Supply similar information and signature for third and subsequent joint inventors.)

French Language Declaration

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Signature de l'inventeur	Date	Fifth inventor's signature	Date
Domicile		Residence	
Nationalité		Citizenship	
Adresse Postale		Post Office Address	
Nom complete du second co-inventeur, le cas echeant		Full name of sixth joint inventor, if any	
Signature de l'inventeur	Date	Sixth inventor's signature	Date
Domicile		Residence	
Nationalité		Citizenship	
Adresse Postale		Post Office Address	

(Fournir les mêmes renseignements et la signature de tout co-inventeur supplémentaire.)

(Supply similar information and signature for third and subsequent joint inventors.)